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(54) Coating process utilising cellulose esters such as cellulose acetate butyrate

(57) A pigmented resinous solvent-based composition also including a cellulose ester such as cellulose acetate butyrate is applied to a metal substrate and allowed to dry at room temperature; a powdered thermosetting clear acrylic, polyester or epoxy resin and cross-linking agent therefor are electrostatically applied and cured by heating.

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SPECIFICATION

Coating process utilising cellulose esters such as cellulose acetate butyrate

This invention relates to the preparation of decorative and protective coatings on metal substrates, in particular on automobile bodies.

It is well known in the automobile industry to apply to an automobile body or part thereof a wet pigmented base coat, and then, without any intermediate baking or stoving, to apply a wet transparent top coat. The base coat and top coat are then stoved together to provide the desired decorative and protective coating. It has also been proposed to apply an aqueous pigmented base coating, and after carrying out at least partial curing of the base coating by an intermediate stoving operation, to apply and cure a particulate top coating.

The present invention uses only a single wet coat: the top coat is in powder form, thus reducing the emission of solvent during coating. No intermediate stoving or curing is required.

According to the present invention, there is provided a process for the application of a coating to a metal substrate, which comprises the steps of (1) applying to the substrate a base coat composition comprising a thermosetting acrylic, alkyd or polyester resin and a cross-linking agent therefor, or alternatively a thermoplastic resin; a cellulose ester; a solvent or solvents; and a pigment; (2) allowing the base coat composition to dry at room temperature to a tack-free surface; (3) applying electrostatically a powdered thermosetting clear acrylic, polyester or epoxy resin and cross-linking agent therefor; and (4) subjecting the substrate and materials applied thereto to heat to cure the electrostatically applied powdered resin and, if present, the thermosetting resin in the base coat.

Suitable thermoplastic resins include polymers and copolymers of acrylic and methacrylic esters, e.g. the Rohm and Haas 'Paraloid' Series A-21, B-66 or B-99.

When a thermosetting resin is used in the base coat composition, it can be a conventional resin or can be in the form of a low molecular weight high solids resin.

While various cellulose esters can be used in the base coat composition, the preferred ester is cellulose acetate butyrate. This assists in controlled removal of the solvent after the base coat composition has been applied, leaving a smooth tack-free surface.

Suitable solvents for use in the base coat composition include, for example, mixtures of solvents selected from xylene, n-butyl acetate, n-butanol and Ektasolve EE Acetate (ethylene glycol monoethyl ether acetate), the last two solvents being used in minor amounts.

The pigments used in the base coat composition may be of various kinds, for example to give straight shades or metallic finishes.

The process of the invention is particularly suitable for use in the manufacture of automobiles, because these are products where frequent colour changes are made. Although the invention uses powder coating to form the top coat, the powder is unpigmented and so the difficulty and expense involved in changing from one coloured powder to another are avoided. Both straight shade and metallic finishes can be obtained using powder as the major component of the coating. Futhermore, recovery of powder is simplified. When metal flake, such as aluminium flake, is used in the production of a metallic finish, the presence of the preferred cellulose ester, cellulose acetate butyrate, optimises the orientation of the flakes in the base coat for the best visual effect.

The electrostatically applied top coat comprises a powdered thermosetting clear acrylic, polyester or epoxy resin, together with a cross-linking agent. Suitable clear powders are commercially available. The acrylic resin may be, for example, of the type having an acrylic backbone incorporating glycidyl methacrylate groups, used with a long chain dicarboxylic acid, e.g. sebacic or dodecanedioic acid, as cross-linking agent. The polyester resin may be a carboxylated polyester, which may be cross-linked with polyfunctional glycidyl compounds such as triglycidylisocyanurate, or a hydroxylated polyester cross-linked with polyfunctional isocyanates such as caprolactam blocked isophoronediisocyanate. Hybrid polyester-epoxy resins may also be used.

The powder preferably has a particle size which is 100% < 100μm, 80% < 50μm and 5% < 10μm.

In order that the invention may be more clearly understood, specific examples thereof will now be given.

Example 1

A base coat composition was prepared from the following materials:

Synedol 2263 XB (a hydroxyl substituted acrylic resin supplied by Synres (U.K.) Limited, as a 50% w/w solution in xylene/n-butanol.

Maprenal MF600 (Hoechst) (cross-linking agent—a butylated melamine derivative);

Cellulose acetate butyrate, type CAB 551-0.2 (Eastman Kodak Company), dissolved in a mixture of butyl acetate and xylene in the proportions of 60:40 by weight;

Stapa Mobilux R167 (non-leafing acid-resistant aluminium flake).

The acrylic resin and cross-linking agent were present in proportions of between 70:30 and 80:20 by weight; the acrylic resin and cellulose acetate butyrate were present in approximately equal proportions by weight. The aluminium flake was present to the extent of 20% w/w of the total solids content, to give

a silver shade.

The viscosity of the base coat composition was adjusted by adding butyl acetate and xylene (60:40 by weight) so that the total solids content was about 15% w/w at a spray viscosity of 15 seconds (Ford Cup No. 4) for hand spraying.

The base coat composition was sprayed on to a steel substrate using successive thin coats to a total thickness of 15 to 20μm, allowing the solvent to flash off at room temperature before applying the next coat.

The material used for the powder top coat was a commercially available clear polyester powder based on Scado P2400, a carboxylated polyester, cross-linked with trisglycidylisocyanurate, having a particle size of 100%>100μm. It was applied electrostatically to a thickness of 45 to 70μm, so that the total coating thickness was 60 to 90μm.

The coated steel substrate was placed in a cold oven, which was heated to a temperature of 180°C over a period of 15 minutes. During this time, the residual solvent in the base coat evaporated. The temperature was then kept at 180°C for a further 20 minutes for curing to take place. The resulting coating was hard and glossy, and had a metallic silver shade.

Example 2

The procedure of Example 1 was repeated, using 15% w/w of the aluminium flake and 15% w/w of a coloured pigment (Irgazin Blue 3GT) in the base coat composition. A hard glossy coating was obtained.

Example 3

The procedure of Example 1 was repeated, using a commercially available clear acrylic powder based on a glycidyl methacrylate copolymer cross-linked with dodecanedioic acid for the powder top coat. The powder, having a particle size of 100%<1000μm, was applied electrostatically to a thickness of 60 to 100μm, so that the total coating thickness was 75 to 120μm. Heating was carried out as described in Example 1, but using an oven set at 170°C. A hard glossy coating was obtained.

Example 4

The procedure of Example 1 was repeated, using as the top coat a commercially available clear polyester powder based on Scado P2504, a hydroxlated polyester, cross-linked with caprolactam blocked isophoronediisocyanate. The oven was set at 180°C. A hard glossy coating was obtained.

Example 5

A base coat composition was prepared from the following materials:

'Paraloid' B-66 (a thermoplastic methyl/butyl methacrylate copolymer);

Cellulose acetate butyrate (CAB 551-0.2);

Titanium dioxide pigment (Tioxide R-TC4);

Modaflow (Monsanto Co.) (flow acid);

Sovent: a mixture of toluene, n-butyl acetate, cyclohexanone, Ektasolve EE Acetate (Eastman Kodak Company), and methyl ethyl ketone in the ratio 20:35:11:16:18 by weight.

The acrylic resin and the cellulose acetate butyrate were present in proportions of between 70:30 and 30:10 by weight. The titanium dioxide level was adjusted to give a pigment volume concentration of 25–27%.

The viscosity of the base coat composition was adjusted with the solvent blend to give a solids content of 25% w/w at a spray viscosity of 15 seconds (Ford Cup No. 4) for hand spraying.

The base coat composition was sprayed on to a steel substrate in successive thin coats to a thickness of 25 to 30 μm, the solvent being allowed to flash off at room temperature after the application of each coat.

The powdered polyester top coat was applied as described in Example 1, to a thickness of 45 to 70μm. The coated steel substrate was heated as described in Example 1. A hard glossy white coating was obtained.

Example 6

The procedure of Example 5 was followed in the preparation and spraying of a base coat composition on to a substrate.

A clear acrylic powder was used for the powder top coat, and was applied electrostatically to a thickness of 60 to 100μm. The coated steel substrate was then heated as described in Example 1 except that the oven temperature was 160–170°C. A hard glossy white coating was obtained.

Another sample of the coated steel substrate was heated by being placed in an oven at 160–170°C for 20 minutes. A hard glossy white coating was obtained.

Example 7

The procedure of Example 5 was followed in the preparation and spraying of a base coat composition on to a steel substrate.

A clear polyester powder, as described in Example 4, was used for the powder top coat. A sample of the coated steel substrate was heated in an initially cold oven to a temperature of 180°C over a period of 15 minutes, then kept at 180°C for a further 20 minutes. Another sample was placed in a oven at 180°C for 20 minutes. Hard glossy white coatings were obtained in each case.

Example 8

A base coat composition was prepared from the following materials:

Synedol 2263 XB (as used in Example 1);

Cymel 300 (Cyanamid) (cross-linking agent—a melamine derivative);

Cellulose acetate butyrate, type CAB

551–0.2 (Eastman [illegible]k Company), dissolved in a mixture of n-butyl acetate and xylene in the proportions of 70:30 by weight; Sparkle Silver 5000 AR (Siberline Ltd) (non-leafing acid-resistant aluminium flake).

The acrylic resin and cross-linking agent were present in proportions of between 70:30 and 90:10 by weight; the acrylic resin and cellulose acetate butyrate were present in approximately equal proportions by weight. The aluminium flake was present to the extent of 20% w/w of the total solids content.

The viscosity of the base coat composition was adjusted as described in Example 1. The remaining procedure of Example 1 was followed, with a similar result. As a cross-linking agent of the Cymel type was used, it was also possible to put the coated substrate directly into an oven at the curing temperature of the particular powder.When this method of curing is used it is preferable to use a powder having delayed curing action, such as hydroxylated polyester cross-linked with caprolactam blocked isophoronediisocyanate, although satisfactory results have been obtained with acrylic powders.

CLAIMS

1. A process for the application of a coating to a metal substrate which comprises the steps of (1) applying to the substrate a base coat composition comprising a thermosetting acrylic, alkyd or polyester resin and a cross-linking agent therefor, or alternatively a thermoplastic resin; a cellulose ester; a solvent or solvents; and a pigment; (2) allowing the base coat composition to dry at room temperature to a tack-free surface; (3) applying electrostatically a powdered thermosetting clear acrylic, polyester or epoxy resin and cross-linking agent therefor; and (4) subjecting the substrate and materials applied thereto to heat to cure the electrostatically applied powdered resin and, if present, the thermosetting resin in the base coat.

2. A process as claimed in Claim 1, wherein the cellulose ester is cellulose acetate butyrate.

3. A process as claimed in Claim 1 or 2, wherein the base coat composition is applied to a thickness of 15 to 20μm.

4. A process as claimed in Claim 1, 2 or 3, wherein the powdered resin is a polyester resin and is applied to a thickness of 45 to 70μm.

5. A process as claimed in Claim 1, 2 or 3, wheren the powdered resin is an acrylic resin and is applied to a thickness of 60 to 100μm.

6. A process as claimed in any one of claims 1 to 5, wherein the powdered resin has a particle size which is 100%<100μm, 80%<50μm, and 5%<10μm.

7. A process as claimed in Claim 1 and substantially as hereinbefore described.

8. A [illegible]l substrate which has been coated by [illegible]process as claimed in Claim 1.

9. A metal substrate which has been coated by a process as claimed in any one of Claims 2 to 6.

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